AR226_3253

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DuPont Haskell Laboratory for Health
and Environmental Sciences

August 5, 2002

cc: G. A. Ladics

MR [redacted]

TO: MR FILE [redacted] POCKET H-24691

FROM: J. C. MASLANKA

TEST SUBSTANCE STABILITY OF [redacted]

Medical Research Project Number: [redacted]
Haskell Sample Number: H-24691
Analytical Test Code: [redacted]
Analytical Report Number: HA-2002-055

Notebook References: [redacted]

Attached is the analytical report for the study identified above. This analysis was performed to satisfy protocol requirements for MR [redacted] but may also be used for other purposes.

Company Sanitized. Does not contain TSCA CBI

TEST SUBSTANCE STABILITY OF [redacted]

Medical Research Project Number: [redacted]
Haskell Sample Number: H-24961
Analytical Report Number: HA-2002-055

SUMMARY

A sample of [redacted] was received May 22, 2002, and analyzed May 30, 2002. The percentage of active ingredient (a.i.) was measured to be 101.8% ± 2.9 with a range of 99.5 to 105.0% for replicate analyses (n = 3). The sponsor reported a purity of [redacted] when sample was submitted.

SIGNATURES:

Analysis by: Richard F. Rossi — 05-Aug-2002
Richard F. Rossi
Chemistry Associate
Date

Report by: Janet C. Maslanka — 05-Aug-2002
Janet C. Maslanka
Senior Staff Chemist
Date

Date issued: 05-Aug.-2002

METHODS

Analysis for the percentage of a.i. in a sample of [redacted] was performed by gas chromatography (GC). Sonication in methanol was required for preparation of test substance, internal standard and analytical standard stock to ensure that [redacted] was completely dissolved.

SAMPLE PREPARATION & ANALYSIS

Aliquots (0.0399, 0.0334, 0.0384 grams) of [redacted] were dissolved in methanol (100 mL) to give nominal concentrations of 399, 334, and 384 ppm. The aliquots were further diluted to a nominal concentration of 23.9, 20.0, 23.0 ppm, respectively, and analyzed according to the following method.

INSTRUMENT & CONDITIONS

Instrument:	Hewlett-Packard Model 6890 GC
Column:	J&W DB-1701, 30 M, 0.32 mm ID, 1.0 μm film thickness
Injector:	Split, 250°C
Detector:	FID; 250°C
Carrier Gas:	Helium (2.1 mL/min)
Split vent:	45.1 mL/min
Injection Volume:	2 microliter
Oven Program:	Gradient
Initial Temperature:	100°C
Initial Time:	1.0 min.
Level 1 Rate:	20°C/min.
Level 1 Temperature:	260°C
Level 1 Time:	0.00 min.
Total run time:	9.00 min.

CALIBRATION & QUANTITATION

c. Calibration and Quantitation

An analytical standard [redacted] was purchased from Fluorochem USA for the analysis. A stock solution was prepared in methanol. This stock solution was sonicated to assure that all material was in solution. Calibration solutions of approximately 10.64 to 42.56 ppm were prepared in methanol. A stock solution of internal standard [redacted] was prepared in methanol and added to each calibration standard and test solution to give a final concentration of 40 ppm. The ratio of the internal standard and [redacted] peak heights from replicate GC analysis of these solutions were used to construct a calibration curve by least squares regression. Measured concentrations for each purity solution were determined by applying the peak height ratios from replicate injections of each sample to the calibration curve.

RESULTS

[redacted] eluted from the GC column as resolved peak with a retention time of approximately 3.4 minutes. The sponsor reported a purity of [redacted] when the sample was submitted.

Table 1. The percent of a.i. in the [redacted] sample analyzed May 30, 2002.

Aliquot	ppm [redacted] Targeted	Measured	Percent Nominal
1	23.9	25.1	105.0
2	20.0	19.9	99.5
3	23.0	23.2	100.9
Average Percent Nominal			**101.8**
Standard Deviation			**± 2.9**
Coefficient of Variation			**3%**